National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Material® 2065

### Ultraviolet–Visible–Near-Infrared Transmission Wavelength/Vacuum Wavenumber Standard

This Standard Reference Material (SRM) is a certified transfer standard intended for the verification and calibration of the wavelength/wavenumber scale of ultraviolet (UV)–Visible–Near-Infrared (NIR) spectrometers operating in transmission mode. In this certificate, spectral features are referred to as *bands* if their location is determined by the center-of-gravity (COG) algorithm, whereas those determined by a five point cubic polynomial fit are referred to as *peak* locations. SRM 2065 is certified for the location of the seven absorbance bands (COG) in the spectral region from 10 300 $cm^{-1}$ to 5 130 $cm^{-1}$ at 4 $cm^{-1}$ resolution. In addition, SRM 2065 is certified for the location of seven absorbance bands in the spectral region from 970 nm to 1 946 nm and 13 additional transmittance peaks spanning the spectral region from 334 nm to 805 nm.

SRM 2065 is a glass consisting of a combination of rare earth oxides of mole fractions 3.00 % holmium oxide ($Ho_2O_3$), 1.30 % samarium oxide ($Sm_2O_3$), 0.68 % ytterbium oxide ($Yb_2O_3$), and 0.47 % neodymium oxide ($Nd_2O_3$) in a base glass containing oxides of lanthanum ($La_2O_3$), boron ($B_2O_3$), silicon ($SiO_2$), and zirconium ($ZrO_2$). The optical filter is 25 mm in diameter and 1.5 mm thick. This combination of the rare earth oxide concentrations and filter thickness yields absorption bands between 0.1 and 0.6 absorbance units in the NIR and 5 % to 60 % transmittance in the UV–Visible.

**Certification:** The certified absorbance/transmittance band locations for SRM 2065 are given in Tables 1, 3, and 5 of this certificate.

**Expiration of Certification:** The certification of this SRM is valid until **31 December 2007**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Instructions for Use). However, the certification is nullified if the SRM is modified or physically damaged.

**Maintenance of SRM Certification:** NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Production and certification of SRM 2065 were performed by S.J. Choquette, L.E. O'Neal, and D.L. Duewer of the NIST Analytical Chemistry Division (ACD). Assistance was provided by L.M. Hanssen, C. Zhu, and E.A. Early of the NIST Optical Technology Division (OTD).

The SRM filters were cut and polished by J. Fuller of the NIST Fabrication Technology Division.

The overall direction and coordination of the technical measurements required for certification of this SRM were performed by S.J. Choquette and G.W. Kramer of the NIST Analytical Chemistry Division.

Statistical consultation was provided by J.J. Filliben and A.I. Aviles of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.W.L. Thomas.

Willie E. May, Chief
Analytical Chemistry Division

Albert Parr, Chief
Optical Technology Division

Gaithersburg, MD 20899
Certificate Issue Date: 28 March 2002

John Rumble, Jr., Acting Chief
Standard Reference Materials Program

**Certified Values:** A NIST certified value represents data reported on an SRM Certificate for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST. The certified [1] vacuum wavenumber locations for the seven absorption bands spanning the range from 10 300 $cm^{-1}$ to 5 130 $cm^{-1}$ are listed in Table 1. These values were obtained at 4 $cm^{-1}$ constant wavenumber resolution and are certified for 25 °C ± 1.5 °C. The absorbance spectrum (NIR) of SRM 2065 is illustrated in Figure 1 of this certificate. Atmospheric water vapor is a significant source of variance for band 3, and this band should be used with caution when calibrating commercial spectrometers with SRM 2065.

The certified values for the NIR air wavelength band locations for the seven absorption bands from 976 nm to 1946 nm for 3 nm spectral resolution at 22 °C ± 2 °C are listed in Table 3.

The certified air wavelength values for the 12 transmittance peaks (334 nm to 645 nm) of SRM 2065 in the UV-Visible are listed in Table 5 for 1 nm spectral bandwidth. The UV–Visible and NIR transmittance spectra of SRM 2065 are shown in Figure 2.

**Reference Values:** A NIST reference value is a best estimate of the true value on a NIST Certificate/Certificate of Analysis/Report of Investigation where not all known or suspected sources of bias have been fully investigated. The reference values [1] for the vacuum wavenumber locations of the seven absorption bands spanning the range from 10 300 $cm^{-1}$ to 5 130 $cm^{-1}$ for six additional spectral resolutions are listed in Table 2. These values are valid for filter temperatures of 25 °C ± 1.5 °C. When using these optical standards to verify the wavenumber scale of a spectrometer, the certified or reference values that are most representative of the spectral bandwidth of the spectrometer being tested should be used. The reference values of the NIR (976 nm - 1946 nm) air wavelength band locations of SRM 2065 at 5 nm and 10 nm spectral resolution are given in Table 4.

The reference values of the temperature coefficients and 0 °C intercepts of the vacuum wavenumber COG band locations of SRM 2065 at 4 $cm^{-1}$ resolution are listed in Table 7. The filter temperature can be a significant source of variance of the NIR band locations. Band locations of the filter were determined using the COG algorithm with a 10 % band fraction over a temperature range between 6 °C and 59 °C. The location of each absorbance band as a function of temperature was determined by a least squares fit to obtain the temperature coefficients given in Table 7.

**Information Values:** A NIST information value is a noncertified value with no uncertainties reported as there is insufficient information to make an assessment of the uncertainties. Information values [1] for the UV–Visible transmittance peak locations of SRM 2065 as a function of spectral resolution are given in Table 6. Information values for the NIR vacuum wavelength temperature coefficients and 0 °C degree intercepts are given in Table 8 for 64 $cm^{-1}$ and Table 9 for 128 $cm^{-1}$ constant wavenumber resolution.

**Uncertainties for Certified and Reference Values:** The expanded uncertainty ($U_{95}$) for the wavenumber and wavelength band locations given in Tables 1 through 5 and 7 are determined from the appropriate combination of component standard uncertainties (i.e., estimated standard deviations), with a coverage factor based on the Student's *t*-distribution, to define the interval within which the unknown value of the band/peak can be asserted to lie with a level of confidence of approximately 95 % [2]. Components of the uncertainty for the NIR include: calibration of the Fourier transform (FT) spectrometers, COG location estimate, location shift due to temperature, and water vapor interference. Components of the uncertainty for the UV–Visible wavelength values include instrumental line shape anomalies, instrument calibration bias, and a component to account for bias between the COG and cubic fit peak location methods.

**Measurement Conditions:** The certification measurements for the NIR spectral region (10 300 $cm^{-1}$ to 5 130 $cm^{-1}$) were made using Bruker IFS66 and Bomem DA FT spectrometers.[1] The Bruker FT spectrometer was calibrated in vacuum wavenumber units using NIST SRM 2517 Wavelength Reference Absorption Cell-Acetylene [3]. The IFS66 spectrometer calibration was validated with ambient water vapor bands. The Bomem spectrometer was calibrated using ambient water vapor and carbon dioxide bands. The dispersive, air wavelength, NIR measurements were performed on a Perkin Elmer Lambda 900 (PE900) spectrometer, a Varian Cary 5E spectrometer, and the NIST OTD Reference Spectrophotometer for Regular Spectral Transmittance. The air wavelength axis of the PE900 spectrometer was calibrated in the NIR using the emission lines of a Kr atomic emission pen lamp and validated using the values for the second order positions of the emission lines of the

[1] Certain commercial equipment, instruments, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

internal $D_2$ source. The Varian Cary 5E was calibrated using the internal $D_2$ source and an external Hg emission lamp.

The certification measurements for the UV–Visible (334 nm to 804 nm) air wavelength band locations were obtained using the PE900 and the NIST ACD National Reference UV–Visible Spectrometer (HAS II) [4]. The HAS II instrument is qualified quarterly for photometric and wavelength calibration. It is wavelength calibrated using Hg and Ne atomic emission pen lamps and validated with SRM 2034 Holmium Oxide Wavelength Solution Standard from 240 nm to 650 nm. The PE900 was wavelength calibrated in the UV–Visible using a Hg emission pen lamp and validated with SRM 2034 and the emission lines of the internal $D_2$ source.

Details of the measurements and data analysis for both the NIR and UV–Visible Measurements can be found in Reference [5].

**Wavenumber and Wavelength Band Location Methodology:** The method used to determine the certified NIR wavenumber ($\upsilon$) and wavelength ($\lambda$) band locations of SRM 2065 is the COG technique [6-8]. If another technique is used, a comparison with the certified values **may not be valid**. In this certificate, positions determined with the COG algorithm are referred to as *band* locations, whereas those determined by a five point cubic polynomial fit to the transmittance minimum, are referred to as *peak* locations. Only those values listed in Tables 5 and 6 (1 nm, 3 nm and 5 nm resolution UV–Visible peak transmittance locations) are peak locations. For COG calculations, a 10 % fraction of the band was used for both wavenumber and wavelength absorption data. Further information on the use of this algorithm with other NIST SRMs can be found in Reference [8]. NIST will provide, upon request, a copy the COG algorithm used for band certification by contacting S.J. Choquette at steven.choquette@nist.gov or by fax at (301) 977-0587.

**Handling and Storage:** To maintain the integrity of SRM 2065, the filter should only be handled in its optical mount. While not in use, the SRM should be stored in the container provided or one with similar or better mechanical protection.

**INSTRUCTIONS FOR USE**

Carefully insert SRM 2065 into the sample beam of the spectrometer being tested. Measurements under a dry nitrogen purge are highly recommended. If a nitrogen purge is not available, the locations of band 1 and band 3 may differ significantly from the certified values. Acquire the absorbance/transmittance spectrum, referenced to air, at a nominal temperature of 25 °C ± 1.5 °C. Compare each measured band location to its certified value listed in the appropriate table (Tables 1, 3, or 5) for the spectral bandwidth most representative of the spectrometer being used. Band locations in Tables 1 and 2 are vacuum wavenumber values, while those in Tables 3 and 5 are air wavelength values. To convert the values in Tables 1 and 2 to air wavenumber, the appropriate correction for the index of refraction of air must be applied [9]. Taking into account the certification uncertainty of each band of SRM 2065, any statistically significant differences between the measured and certified band locations may then be used to recalibrate the spectrometer wavenumber/wavelength scale.

REFERENCES


[1] May, W.E., Parris, R.M., Beck II, C. M., Fassett, J.D., Greenberg, R.R., Guenther, F.R., Kramer, G.W., Wise, S.A., Gills, T.E., Gettings, R.J., and MacDonald, B.S., "Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurement," NIST Special Publication 260-136, (2000).

[2] Guide to the Expression of Uncertainty in Measurement, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor B.N. and Kuyatt, C.E., " Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC, (1994); available at http://physics.nist.gov/Pubs.

[3] Gilbert, S.L. and Swann, W.C., "Standard Reference Materials: Acetylene $C_2H_2$ Absorption Reference for 1510 nm – 1540 nm Wavelength Calibration-SRM 2517," NIST Special Publication 260-133, (1997).

[4] Mavrodineanu, R., "An Accurate Spectrophotometer for Measuring the Transmittance of Solid and Liquid Materials," NBS J. Res. Vol. 76A, No. 5, pp. 405-425, (1972).

[5] Choquette, S.J., Duewer, D.L., and O'Neal L.E., "Standard Reference Material® 2065: X-axis Variability in Fourier Transform Near Infrared Spectroscopy," (publication in preparation).
[6] Cameron, D.G., Kauppinen, J.K., Moffat, J.K., and Mantsch, H.H., "Precision in Condensed Phase Vibrational Spectroscopy," Appl. Spectrosc., Vol. 36, pp. 245-250, (1982).
[7] Zhu, C. and Hanssen, L.M., "Studies of Polystyrene Wavenumber Standard for Infrared Spectrometry," 11th International Conference of Fourier Transform Spectrometry, AIP proceedings, 430, Ed. J.A. de Haseth, pp. 491-494, American Institute of Physics, NY, (1998).
[8] ASTM E 1421-99, "Standard Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) spectrometers: Level Zero and Level One Tests," Annual Book of ASTM Standards 2001, Vol. 03.06, Section Three, Analytical Procedures, pp. 513-524, (2001).
[9] Edlen, B., "The Refractive Index of Air," Metrologia, Vol. 2, No. 2, pp. 71-80, (1966).

*Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.*

Table 1. Certified[1] Vacuum Wavenumber Band Locations[3] of SRM 2065 and Uncertainties[4] at 4 $cm^{-1}$ Resolution

| Resolution ($cm^{-1}$) | $B_1$ ($cm^{-1}$) | $B_2$ ($cm^{-1}$) | $B_3$ ($cm^{-1}$) | $B_4$ ($cm^{-1}$) | $B_5$ ($cm^{-1}$) | $B_6$ ($cm^{-1}$) | $B_7$ ($cm^{-1}$) |
|---|---|---|---|---|---|---|---|
| 4 | 5139.3 ± 0.5 | 6806.3 ± 0.9 | 7314.9 ± 0.7 | 8180.1 ± 0.9 | 8682.6 ± 1.3 | 9294.4 ± 0.8 | 10245.6 ± 0.6 |

Table 2. Reference[2] Vacuum Wavenumber Band Locations[3] of SRM 2065 and Uncertainties[4] for Given Resolutions

| Resolution ($cm^{-1}$) | $B_1$ ($cm^{-1}$) | $B_2$ ($cm^{-1}$) | $B_3$ ($cm^{-1}$) | $B_4$ ($cm^{-1}$) | $B_5$ ($cm^{-1}$) | $B_6$ ($cm^{-1}$) | $B_7$ ($cm^{-1}$) |
|---|---|---|---|---|---|---|---|
| 2 | 5139.3 ± 0.5 | 6806.3 ± 0.9 | 7314.9 ± 0.7 | 8180.1 ± 0.9 | 8682.6 ± 1.3 | 9294.4 ± 0.8 | 10245.6 ± 0.6 |
| 8 | 5139.3 ± 0.5 | 6806.3 ± 0.9 | 7314.9 ± 0.7 | 8180.1 ± 0.9 | 8682.7 ± 1.3 | 9294.4 ± 0.8 | 10245.6 ± 0.6 |
| 16 | 5139.5 ± 0.5 | 6806.4 ± 0.9 | 7315.0 ± 0.7 | 8180.1 ± 0.9 | 8683.0 ± 1.3 | 9294.4 ± 0.8 | 10245.3 ± 0.6 |
| 32 | 5139.8 ± 0.5 | 6806.7 ± 0.9 | 7315.0 ± 0.7 | 8180.4 ± 0.9 | 8684.2 ± 1.3 | 9294.6 ± 0.8 | 10244.5 ± 0.6 |
| 64 | 5140.2 ± 0.5 | 6807.8 ± 0.9 | 7315.3 ± 0.7 | 8181.7 ± 0.9 | 8687.3 ± 1.4 | 9294.9 ± 0.8 | 10243.2 ± 0.6 |
| 128 | 5136.2 ± 1.0 | 6810.0 ± 0.9 | 7314.3 ± 2.0 | 8185.8 ± 1.1 | 8690.4 ± 1.4 | 9297.1 ± 0.8 | 10246.4 ± 2.0 |

[1] A NIST certified value represents data reported on an SRM Certificate for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST. The 4 $cm^{-1}$ constant wavenumber band locations are certified. All other band locations as a function of spectral resolution should be considered reference values only.

[2] A NIST reference value is a best estimate of the true value on a NIST Certificate/Certificate of Analysis/Report of Investigation where not all known or suspected sources of bias have been fully investigated.

[3] Band location determined using a Center-of-Gravity method with a band fraction of 0.1; see Figure 1 for band identification.

[4] Uncertainties represent $U_{95}$, the expanded uncertainty calculated in accordance with Reference [2].

Table 3. Certified Air Wavelength Band Locations[1] for SRM 2065
and Uncertainties at 3 nm Spectral Bandwidth

| Band | 3 nm Spectral Bandwidth Band Location[2] (nm) |
|---|---|
| 7 | 975.9 ± 0.3 |
| 6 | 1075.7 ± 0.2 |
| 5 | 1151.4 ± 0.1 |
| 4 | 1222.2 ± 0.4 |
| 3 | 1366.7 ± 0.4 |
| 2 | 1469.0 ± 0.4 |
| 1 | 1945.5 ± 0.3 |

Table 4. Reference Air Wavelength Band Locations[3] for SRM 2065
and Uncertainties for Given Spectral Bandwidths

| Band | 5 nm Spectral Bandwidth Band Location (nm) | 10 nm Spectral Bandwidth Band Location (nm) |
|---|---|---|
| 7 | 976.0 ± 0.2 | 976.0 ± 0.6 |
| 6 | 1075.8 ± 0.9 | 1075.9 ± 2.2 |
| 5 | 1151.3 ± 1.0 | 1151.1 ± 3.4 |
| 4 | 1222.2 ± 0.3 | 1222.2 ± 0.9 |
| 3 | 1366.8 ± 0.5 | 1367.1 ± 0.2 |
| 2 | 1469.1 ± 1.7 | 1469.2 ± 3.7 |
| 1 | 1945.5 ± 0.7 | 1945.6 ± 1.5 |

[1] A NIST certified value represents data reported on an SRM Certificate for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST.
[2] Uncertainties represent $U_{95}$, the expanded uncertainty calculated in accordance with Reference [2].
[3] A NIST reference value is a best estimate of the true value on a NIST Certificate/Certificate of Analysis/Report of Investigation where not all known or suspected sources of bias have been fully investigated.

Table 5. Certified Air Wavelength Peak Locations[1] of SRM 2065
and Uncertainties at 1 nm Spectral Bandwidth

| Peak | Average Peak Position[2] (nm) |
|---|---|
| 20 | 334.6 ± 0.1 |
| 19 | 345.4 ± 0.2 |
| 18 | 360.8 ± 0.2 |
| 17 | 374.5 ± 0.1 |
| 16 | 386.1 ± 0.1 |
| 15 | 402.5 ± 0.2 |
| 14 | 417.9 ± 0.1 |
| 13 | 485.4 ± 0.1 |
| 12 | 537.7 ± 0.5 |
| 11 | 583.4 ± 0.3 |
| 10 | 642.4 ± 0.6 |
| 9 | 747.7 ± 0.4 |
| 8 | 804.3 ± 0.5 |

Table 6. Information Air Wavelength Peak Locations[3] of SRM 2065
at 3 nm and 5 nm Resolution

| Peak | Spectral Bandwidth | | |
|---|---|---|---|
| | 1nm | 3nm | 5nm |
| 20 | 334.6 | 334.3 | 334.3 |
| 19 | 345.4 | 345.6 | 346.3 |
| 18 | 360.8 | 361.2 | 361.6 |
| 17 | 374.5 | N/D | N/D |
| 16 | 386.1 | N/D | N/D |
| 15 | 402.5 | 402.4 | 402.7 |
| 14 | 417.9 | 418.0 | 418.3 |
| 13 | 485.4 | 485.3 | 485.5 |
| 12 | 537.7 | 537.9 | 538.3 |
| 11 | 583.4 | 583.4 | 583.4 |
| 10 | 642.4 | 642.5 | 642.9 |
| 9 | 747.7 | 747.7 | N/D |
| 8 | 804.3 | 804.3 | 804.3 |

[1] A NIST certified value represents data reported on an SRM Certificate for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST.
[2] Uncertainties represent $U_{95}$, the expanded uncertainty calculated in accordance with Reference [2].
[3] A NIST information value is a noncertified value with no uncertainties reported as there is insufficient information to make an assessment of the uncertainties.

Table 7. Reference Values[1] for Temperature Coefficients and Intercepts for Vacuum Wavenumber Band Locations at 4 $cm^{-1}$ Resolution

| Band | Temperature Coefficient[2] ($cm^{-1}$/°C) | 0 °C Intercept ($cm^{-1}$) |
|---|---|---|
| 1 | -0.0517 ± 0.0010 | 5140.64 ± 0.04 |
| 2 | 0.0912 ± 0.0004 | 6804.01 ± 0.03 |
| 3 | 0.006 ± 0.014 | 7314.84 ± 0.48 |
| 4 | 0.0619 ± 0.0011 | 8178.45 ± 0.04 |
| 5 | -0.0419 ± 0.0011 | 8683.61 ± 0.07 |
| 6 | -0.0783 ± 0.0009 | 9296.41 ± 0.08 |
| 7 | 0.0191 ± 0.0006 | 10245.15 ± 0.03 |

Table 8. Information Values[3] for Temperature Coefficients and Intercepts for Vacuum Wavenumber Locations at 64 $cm^{-1}$ Resolution

| Band | Coefficient ($cm^{-1}$/°C) | 0 °C Intercept ($cm^{-1}$) |
|---|---|---|
| 1 | -0.085 | 5142.53 |
| 2 | 0.085 | 6806.35 |
| 3 | 0.003 | 7315.64 |
| 4 | 0.069 | 8181.11 |
| 5 | -0.073 | 8690.86 |
| 6 | -0.075 | 9297.66 |
| 7 | 0.022 | 10242.63 |

Table 9. Information Values for Temperature Coefficients and Intercepts for Vacuum Wavenumber Band Locations at 128 $cm^{-1}$ Resolution

| Band | Coefficient ($cm^{-1}$/°C) | 0 °C Intercept ($cm^{-1}$) |
|---|---|---|
| 1 | -0.22 | 5141.59 |
| 2 | 0.073 | 6807.15 |
| 3 | -0.055 | 7312.24 |
| 4 | 0.084 | 8185.24 |
| 5 | N/D | N/D |
| 6 | -0.082 | 9300.25 |
| 7 | 0.025 | 10248.71 |

[1] A NIST reference value is a best estimate of the true value on a NIST Certificate/Certificate of Analysis/Report of Investigation where not all known or suspected sources of bias have been fully investigated.

[2] Uncertainties represent $U_{95}$, the expanded uncertainty calculated in accordance with Reference [2].

[3] A NIST information value is a noncertified value with no uncertainties reported as there is insufficient information to make an assessment of the uncertainties.

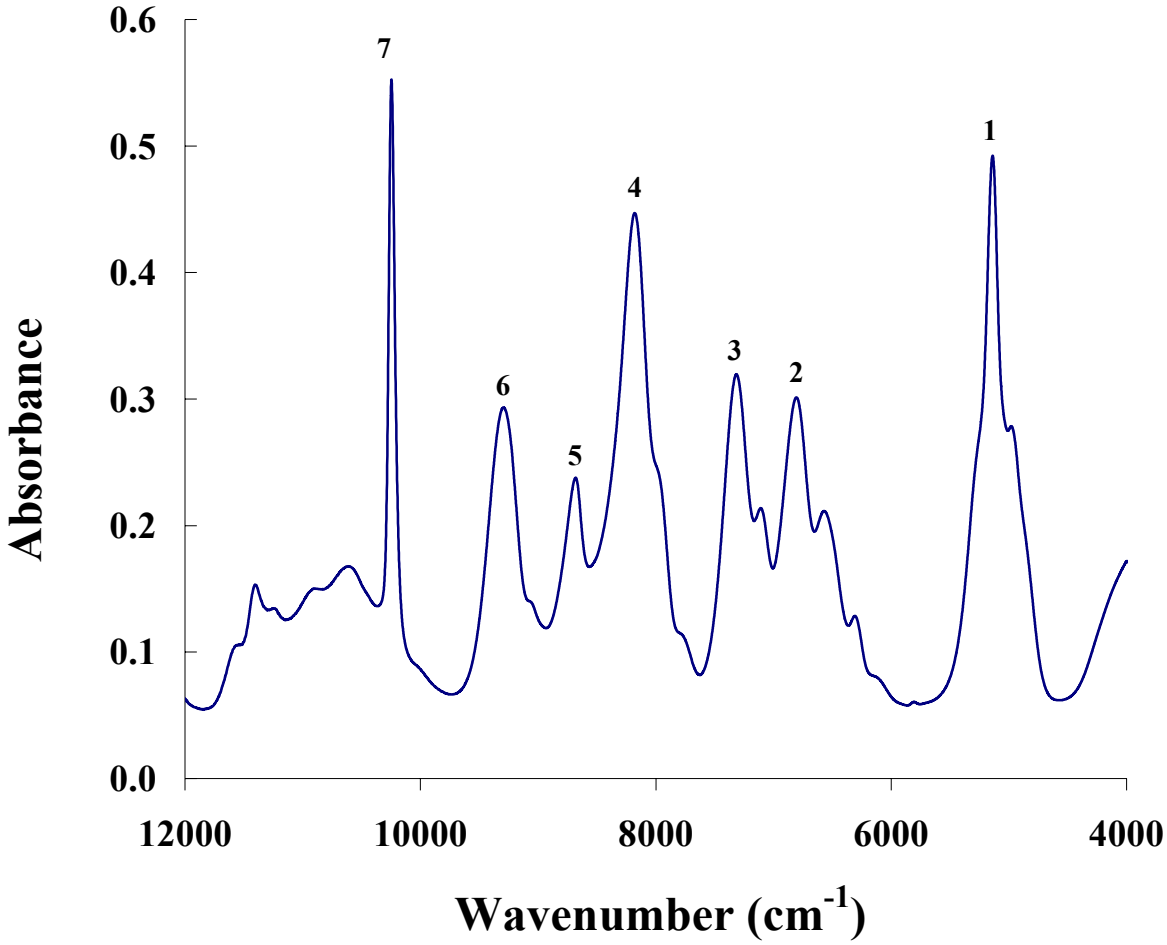


Figure 1. NIR Absorbance Spectrum of SRM 2065 with the band locations indicated.

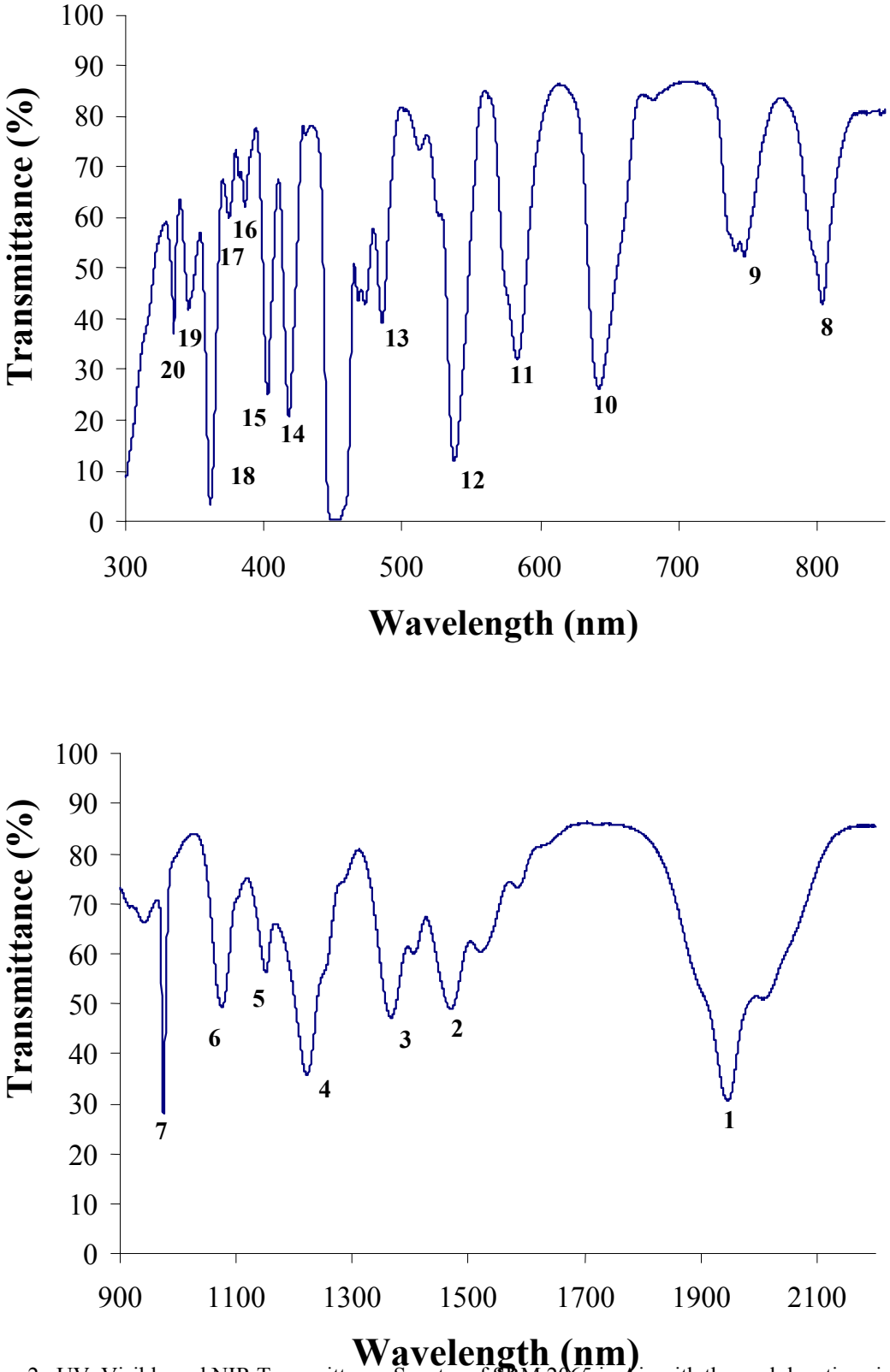


Figure 2. UV–Visible and NIR Transmittance Spectra of SRM 2065 in Air with the peak locations indicated.